

## DYNATECH CORPORATION

### THERMOPHYSICAL PROPERTIES OF HOT PRESSED BERYLLIUM

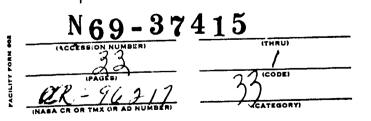
Prepared by: R. P. Tye

### Submitted to:

GENERAL ELECTRIC COMPANY Re-Entry Systems Department 3198 Chestnut Street Philadelphia, Pennsylvania 19101

NAS1-6039

July 1, 1968





Progress through Research



# THERMOPHYSICAL PROPERTIES OF HOT PRESSED BERYLLIUM

Prepared by: R. P. Tye

### Submitted to:

GENERAL ELECTRIC COMPANY Re-Entry Systems Department 3198 Chestnut Street Philadelphia, Pennsylvania 19101

July 1, 1968

Dynatech Report No. 796
Dynatech Project No. GES-4
General Electric P. O. No. 037-£10234

DYNATECH CORPORATION 17 Tudor Street Cambridge, Massachusetts 02139



## TABLE OF CONTENTS

Section		Page
1	INTRODUCTION	1
2	DETAILS OF SAMPLES	2
3	DETAILS OF TESTS	5
	3.1 Thermal Conductivity 3.2 Specific Heat	<b>5</b> 8
	3. 2. 1 Adiabatic Calorimeter Tests 3. 2. 2 Drop Calorimeter	9 11
4	RESULTS AND DISCUSSION	15
	4.1 Thermal Conductivity and Electrical Resistivity 4.2 Specific Heat	15 17
5	SUMMARY	18
	REFERENCES	19





### Section 1

### INTRODUCTION

This report is submitted on the completion of the work carried out at the Thermatest Measurements Laboratory of Dynatech R & D Company for General Electric Company, Re-entry-Systems Department, Philadelphia, Pennsylvania. This work, monitored by Mr. J. Brazel of General Electric Company formed part of a large program covering the heat transfer characteristics of a space vehicle on re-entry into the atmosphere.

The purpose of the investigation was to undertake accurate measurements of the thermal conductivity and specific heat of a number of samples of hot pressed beryllium to temperatures in excess of 900 K. The samples used were cut, in both the parallel to and perpendicular to pressing direction, from the pieces used for the actual construction of the component parts of a measurements space vehicle. This vehicle was constructed for the sole purpose of measuring heat transfer characteristics of a body on re-entry into the earth's atmosphere prior to burning-up.

Since very great effort and care had been taken in the design, development, construction and instrumentation of the device it was desired that the thermophysical properties values of the different pieces of beryllium used should be measured as accurately as possible in order to make the subsequent analysis of heat transfer data as meaningful as possible. Any possible difference or variation of property value due to differences in composition, anisotropy or other variable could be allowed for in the calculations if the actual constructional materials had been evaluated.

The thermal conductivity measurements were carried out using the method of longitudinal heat flow in a rod surrounded by a matched temperature gradient guard tube. The specific heat determinations were made by a combination of adiabatic and drop calorimetry techniques.

In all, measurements of thermal conductivity were carried out on twelve samples over the approximate temperature range 300 to 1300K and of specific heat on thirteen samples over the range 300 to 1100K. Electrical resistivity measurements were also included on some of the thermal conductivity samples.



### Section 2

### DETAILS OF SAMPLES

The various samples were stated to be cut from stocks of not pressed beryllium manufactured by Brush Beryllium Company. Table I contains the stated chemical compositions and densities of each of the stocks used.

Table I

Details of Stock Materials Used

			Cł	emical	Composi	tion WG	%		Other	Density
Lot	Be	BeO	C	Fe	Al	Mg	Si	Mn	Metallic	l "n
3840	98.27	1.78	0.13	0.13	0.11	0.01	0.04	0.01	0.01	1.86
4483	98.3	1.7	0.15	0.14	0.08	0.01	0.04	0.01	0.04	1.85
4811	98.41	1.64	0.12	0.12	0.09	0.01	0.03	0.01	0.04	1.86
4814	98.46	1.60	0.12	0.12	0.09	0.01	0.03	0.01	0.04	1.86
4921	98.4	1.8	0.15	0.13	0.09	0.01	0.03	0.01	0.04	1.86
5085	98.2	1.7	0.12	0.13	0.12	0.03	0.04	0.01	0.04	1.853

3840 was stated to have come from rod stock while the others were from 5200 block.

The thermal conductivity samples were in the form of a has approximately 100 mm long and 13 mm in diameter with tapered ends. At the center of one tapered end there was a hole approximately 6 mm diameter and 25 mm deep drilled to accommodate a tubular pencil heating element. Over the central 45 mm length four small holes approximately 0.2 mm diameter and 2 mm deep were drilled for thermocouple attachement.

The samples for specific heat measurements by adiabatic calorimeter, were in the form of square slabs approximately 63 mm square by 12 mm thick. Holes 3mm diameter were drilled at the corners of the central 38 mm square section.

The sample for drop calorimetry used in the form of cylinders approximately 10 mm diameter and 50 mm long.

The identification of each sample and its relation to the particular spacecraft part are given in Table II.



# DYNATECH

Table II

Dynatech	Test Sample N	umber		
Thermal Conductivity	Adiabatic Calorimetry	Drop Calorimetry	Lot No.	Spacecraft Part
	1, 4	4, 5	3840	EDM P1
	5, 8, 11	2, 3	4483	EDM P2 - P7
9,12 parallel 10,11 perpendicular	2, 6	11, 12	4811	S/C # 1 P1/
6,7,8	3,12,13	8, 9, 10	4814	S/C # 1 P2 - P7
1,2,3	7,9,10	1, 6, 7	4921	S/C # 2 P2 - P7
4,5	~-		5085	S/C # 1 Replacement P 4

On receipt all samples were weighed and their dimensions measured accurately. From these figures the densities of the samples were found to agree very closely with those stated for the different lot numbers.

As a provisional check in the uniformity of the material, electrical resistivity, measurements were carried out at room temperature on each of the thermal conductivity samples. Each sample was fitted with current electrodes at each end and then laid across knife edges at a given accurately measured distance apart, corresponding to the central 40 mm. A steady d. c. current was passed through the sample and the potential differences across the knife edges and a calibrated 0.001 ohm resistance, in series with the sample, measured on a potentiometer. The current was reversed in order to eliminate any thermal voltages and the potential differences measured again. The electrical resistivity was evaluated in terms of the ratio of the measured potential differences and the dimensions of the test sample.

Table III contains values of electrical resistivity obtained for the twelve samples.



Sample No.	Lot No.	$10^6 \rho$ , ohm cm
1	4921	4.28
2	**	4.29
3	11	4.32
4	5085	4.42
5	11	4.39
6	4814	4.54
7	11	4.50
8	11	4.27
9	4811	4.70
10	**	4.59
11	**	4.64
12	11	4.69

These results indicate that some small differences in property could be expected but that with the exception of sample number 8 the property for each batch would not vary significantly. In addition the results for samples 9 to 12 indicate that any anisotropy in property is less than 2%.



### Section 3

### DETAILS OF TESTS

### 3.1 Thermal Conductivity

The tests were performed in the Dynatech axial rod test, an instrument developed specially for use over the approximate temperature range 300 to 1300 K and using the principles of steady state guarded longitudinal heat flow. The apparatus is shown schematically in Figure 1, and a similar one for operation at temperatures up to 870K has been described in detail previously. (1, 2)

The test sample is assembled vertically with its lower tapered end fitting tightly into a large nickel heat-sink block. The upper tapered end contains a snug fitting small pencil heater element and is covered with a nickel isothermal cap fitting tightly over the uppersample taper. The taper jets are used at each end to minimize thermal contact resistance between the tests thus helping to attain a more even temperature distribution. A small thermocouple bead is pegged into each of the small holes along the central section. The thermocouples are made from 0.2 mm wires (either chromel/alumel and platinum/ platinum 10% rhodium materials are used) insulated in twin bore silica or aluminum sheaths in order to determine the temperature gradient along the sample during test. Each thermocouple is fixed so that the wire adjacent to the junction is in an isothermal zone and then tied along the lower length of the sample and brought through a slot, cut into the heatsink, to the lower connection in the base plate.

The sample is surrounded by a nickel guard tube assembly also having small holes for thermocouples in the top and along its length. This assembly is bolted firmly to the heat sink which also contains a thermocouple to insure that the lower extremity of the guard tube operates at the sink temperature, which can be adjusted using a wire wound ceramic core resistance heating element inserted into holes drilled through the sink. A closely wound resistance wire heater formed between two grooved ceramic sleeves is clipped firmly to the top of the guard tube.

Small vertical holes are drilled into the top of the guard tube cap and sample heater cap respectively and two thermocouples attached to each and brought through a small slot cut in the top of the guard tube cap. One pair of thermocouples is used for direct measurement of the temperature at the top of the guard tube and sample cap and the other



two are connected differentially. The output of this thermocouple is connected to a controller which adjusts the power fed to the guard heater in order to maintain the temperature of the guard and sample caps to be the same during the test.

The whole assembly is surrounded by a further cylindrical wire wound heating tube. The space between sample and guard tube is filled carefully with a thermal insulating powder of very low thermal conductivity and the complete assembly is surrounded by further tube and the whole filled with insulation powder. A bell jar covers the assembly and the apparatus evacuated to a pressure of  $10^{-4}$  torr and then backfilled with an inert gas (argon or helium are used).

In operation a steady heat flux is established down the sample by means of constant d. c. electrical input to the heater. The overall temperature of the system is maintained by suitable adjustment of the outer cylindrical heater and the auxiliary heater in the heatsink. The guard heater is controlled automatically by means of the output of the differential thermocouple so that the temperature distribution in the guard and sample caps are the same. This minimizes any radial heat loss from the sample heater to the surroundings so that the measured heat input to the sample heater as measured using a precision resistor network, appears as a heat flux down the sample. Moreover with the sample and guard tube temperatures matched at each end, the temperature gradient along the guard tube is very close to that along the sample, thereby minimizing radial heat losses in this area. The temperature gradient along the guard tube is measured by means of the thermocouples fixed to it and from the overall temperature distribution a correction can be made for any radial losses which may occur.

At equilibrium conditions the sample thermal conductivity is determined as follows

$$\lambda = \frac{q/A}{dT/dx}$$

where

q is the electrical heat input to the heater

A is the cross sectional area of the sample normal to heat flow

dT/dx is the temperature gradient in the test sample

# DYNATECH

In this investigation measurements were carried out by raising the sample temperature in approximate regular increments in the approximate temperature range 300 to 1300K where possible a repeat measurement at some intermediate temperature was made after attaining a temperature in excess of 1200K in order to see if any changes had occurred or whether any contamination had taken place. Following this attempts were made to run the sample to as high a temperature as possible. In practice heater failures and contamination prevented any meaningful results being obtained above 300K. Tests were carried out in different inert atmospheres including argon and helium.

During the course of the test, if the temperature gradients in the sample and guard were not matched exactly, measurements were made and a correction applied for any possible heat lost or gained by the sample. Details of this correction and it application have been outlined in detail in a previous report. In practice, it was found that the correction to be applied was never an excess of 2% and from the majority of the data points it was well within 1%.

Similarly a small correction could be applied for any slight differences in temperature between the sample cap and guard cap. The control of the temperatures was such that this correction was never in excess of  $\Gamma_{ij}^{ij}$ .

For electrical conductors the measured thermal conductivity ( $\lambda$ ) is a summation of several different induction modes, the predominant ones being due to the electrons ( $\lambda_e$ ) and to the lattice imperfections ( $\lambda_g$ ). For electrical conduction  $\lambda_e$  is related closely to the electrical resistivity—by the Wiedemann-Franz Law

$$\lambda_e = \frac{LT}{\rho}$$

where L is the theoretical value of the Lorenz function and T is the absolute temperature. Thus by measuring  $\lambda$  and  $\rho$  during the same experiment it is possible to separate out the two major contributions to the total conduction and be in a position to investigate their behavior in more detail. A significant additional reason for measuring the electrical resistivity is that its evaluation does not involve directly the measurement of temperature and thus any real change found in thermal conductivity due to heat treatment or other causes or apparent changes caused by thermocouple contaminations can be verified very simply.

## DYNATECH

Measurements of electrical resistivity were made on six of the samples over the same temperature range. The samples were chosen on the basis of the results obtained earlier and given in Table III.

Each sample was fitted with a stainless steel electrode on the top cap, the other electrode being connected to the heat sink. The sample was connected in series with a calibrated fixed resistance of 0.001 ohm and a constant 24 Vd. c. supply. During the course of the thermal conductivity measurements either immediately after measurements of conductivity or at convenient temperature chosen to define a complete curve a steady current was applied and the potential drop across the sample was compared with that across the shunt. In each case the current was reversed to eliminate thermal voltages. The "like" arms of the top and bottom thermocouples in the sample were used in order to define the potential length used in there measurements. Results were obtained on all six samples up to approximately 1300K but either sample heater failure or contamination of the sample prevented any reliable measurements being obtained above that temperature.

### 3.2 Specific Heat

These measurements were required with an accuracy of  $\pm$  3% or better if at all possible. In order to attain this objective it was decided to use a combination of adiabatic and drop calorimetry to cover the complete temperature range with a suitable overlap of temperature where techniques could be used.

Thus for the approximate range 300 to 670K the Dynatech Quantitative Thermal Analyser was used and for the approximate range 570 to 1100K the Dynatech SHDW-R20 Drop Calorimeter, with a calibrated copper block receiver, was used. The precominant reason for this choice was based upon the fact that a high accuracy of measurement can be obtained by the adiabatic calorimeter method at temperatures up to 700K, but it becomes potentially less accurate at higher temperatures due to the effects of radiation heat transfer and its heat loss effects while conversely the drop calorimeter, due to the larger total enthalpy changes involved with drops involving large temperature differences between sample and receiver, becomes potentially more accurate at higher temperatures.



### 3.2.1 Adiabatic Calorimeter Tests

The instrument and method including a detailed analysis has been described previously  $^{(3)}$  and only an outline of the method will be given here.

Each sample is weighed accurately and then clamped tightly into a copper container of similar dimensions. The surface of the container is wound uniformly with a a shealthed electrical heating wire and the complete exterior was gold plated and polished. The loaded container is then suspended inside a massive nickel plated and polished copper cylindrical jacket complete with a lid. The jacket and lid contains a uniformly wound sheated electrical heater in intimate contact with the copper for good heat transfer. This container is then mounted upon three small ceramic pegs, inside a series of further radiation shields surrounded by an insulated jacket and then curved with a bell jar.

A four junction differential thermocouple, constructed from 0.3 mm copper constantan wires, consisting of two junctions fixed tightly in the sample container and two similarly fixed in the jacket is used for power control purposes. Similar single thermocouples are fitted to the sample container and jacket but electrically indicated from the differential couple in order to monitor the absolute temperatures of each and one of those in the sample container is connected to a sensitive millivolt recorder. The sample heater is connected to a regulated precision d.c. power supply and the jacket heater to a silicon controller rectifier a.c. power supply controlled by the proportional controller with automatic reset and rate-time action which is actuated by the differential thermocouple.

The whole assembly is evacuated to below  $10^{-4}$  torr to reduce convective and gas conduction heat transfer cooled and allowed to stabilize at some temperature below the lowest mean temperature at which results are required. At equilibrium, power is supplied to the sample container heater at a stable controlled into such that the of temperature is not in excess of  $2 \, \text{deg}^{-1}$ . The power to the jacket heater is controlled automatically by the differential couple controller system so that the temperature of the jacket and sample are the same, thus ensuring that adiabatic conditions are maintained and that there is negligible heat transfer from the sample to its surroundings.

Regular observations of the power input to the heater and of the temperatures of sample and guard tubes and a continuous record of the variation of the rise in temperature



of the sample container with time obtained on the recorder. From these observations a continuous record of the total enthalpy change with temperature is obtained. Prior to any measurements on a test sample, the sample container is measured empty but under similar conditions and over the same temperature range. The specific heat of the test sample can then be derived from the knowledge of its weight and the difference in enthalpy changes between the container with sample and the container alone as follows.

For container alone

$$q = M C_p \frac{d T}{d \tau}$$

where

M is the mass

C<sub>n</sub> the specific heat

 $\frac{d T}{d \tau}$  is the ration use of temperature with time

For container and sample

$$q = M C_{p \text{ heater}} + M C_{p \text{ sample}} \frac{dT}{d\tau}$$

M C p heater is determined as a function of temperature from the calibration runs and evaluating d  $T/d\tau$  from the slope of the recorder trace which is d(emf)/d i.e.

$$\frac{d T}{d \tau} = \frac{d \text{ (emf)}}{d \tau} \frac{d \text{ (emf)}}{T}$$

where d (emf)/T is the thermocouple emf - temperature characteristic obtained from a curve of the appropriate emf  $\underline{v}$  temperature values.

Thus the sample specific heat can be obtained from the curve of  $dT/d\tau$  for sample container and sample length with the derived values of M  $C_{\ p\ heater}$ . Although a continuous record can be obtained, in practice the specific heat is evaluated at a number of discrete temperatures coinciding with appropriate values of the thermocouple emf. Additional values can always be evaluated at any number of intermediate



temperatures should the different specific heat change markedly over a limited temperature range.

In the present work measurements were undertaken on all samples up to approximately 630K and to 670K in some instances. After each measurement the sample weight was checked carefully but no change was found for any sample.

### 3.2.2 Drop Calorimeter

The principle and operation of drop calorimeters of both the ice mantle and calibrated copper receiver varieties are well known<sup>(4,5)</sup> and the instrument used follows the general accepted principles very closely.

The instrument consists basically of a uniformly wound electrically heated multizone controlled temperature, tubular furnace mounted vertically above a large copper receiver. The copper receiver is surrounded by an insulation jacket and the whole is supported inside a large container of water held at a near constant temperature. Between the lower end of the furnace and the mouth of the receiver is a water cooled internally polished cylinder 75 mm diameter and 150 mm long with a moveable cover with polished surfaces at each end, the lower one serving as the lid of the copper receiver. A liquid cooling coil surrounds the zone and water controlled at the same temperature as that surrounding the receiver is circulating through it.

The reciever is a block of copper 100 mm external diameter and 150 mm long, with a well 40 mm diameter and 100 mm deep. Inserted into various wells drilled at different heights in the walls of the receiver are ten thermocouple junctions connected to form a thermopile in order to accurately measure the temperature of the receiver.

Two 0.6 mm diameter wire, chromel alumel thermocouples are placed at two positions in the central 50 mm section of the furnace tube, one serving to activate a controller to maintain the temperature constant to better than 1K and both being used for absolute temperature measurements when necessary  $\rho$ rior to a "drop".

In operation a sample is weighed accurately and then suspended with a 0.3 mm support wire of a nickel-aluminum alloy at the center zone of the furnace. The free end of the wire passing over a free moving pulley at the top of the furnace with its end fixed to

# DYNATECH

a nylon plunger which can slide freely up and down an external low friction track. The wire is so arranged that the sample is suspended vertically along the center line of the furnace and of such a length that when the plunger comes to rest at the end of its travel the sample rests upon the base of the wall in the receiver. Great care is taken to ensure that the sample can fall quickly and freely without hitting the sides of the furnace tubes or the intermediate cylindrical protection tube and decelerates over the last 20 mm of its fall.

The sample is allowed to attain the equilibrium temperature for a period of time usually the order of 1 - 2 hr and regular readings of the temperature measurement thermocouples taken. During the thirty minutes prior to a "drop" the temperature of the receiver has noted every 30 s. At a given time when the sample temperature is deemed constant and the very small drift in temperature of the receiver has been noted, the sample is dropped quickly. As the sample is dropped the radiation shields covering the intermediate zone are moved sideways to allow the sample to fall and come to rest in the receiver. When the sample has come to rest, these shields are quickly returned to the original position in order to reduce any radiation transfer of heat from the turnace to the receiver or convective and radiant heat transfer from the receiver to the outside.

The temperature of the receiver is taken regularly at 30s intervals for the first thirty minutes following the drop followed by 60s intervals for the next thirty to sixty minutes in order to determine the rise in temperature and the subsequent drift in temperature of the receiver. Following a drop, the receiver system is allowed to come to equilibrium for the order of two hours and a further drop made on the same or a different sample.

Prior to any measurements on the test sample the copper receiver was calibrated by use of an electrical resistance heater of very small mass, compared to the receiver. Three individual measurements were made and the results agreed to better than 0.8%.

In an adiabatic system the enthalpy loss of a sample initially at a high temperature must be equal to the enthalpy gain of all other parts of a system at lower temperatures. The basic part of the system is, in this case, a massive copper receiver and thus providing heat losses are eliminated the enthalpy loss of the sample is equal to that gained by the receiver.



Thus

$$\Delta$$
 H sample =  $-\Delta$ H receiver 
$$(T_i - T_f) \qquad T_o - T_f$$
 = M C<sub>p</sub>  $(T_o - T_f) - m$  C'<sub>p</sub>  $(T_i - T_f)$ 

where  $T_i$ ,  $T_o$  and  $T_f$  are initial temperatures of sample and receiver and final equilibrium temperature of receiver respectively and M  $C_p$  is the heat capacity of the receiver and m  $C_p$  is the heat capacity of the drop wire.

Since 
$$T_f > T_O$$

$$\Delta H \text{ sample} = M C_p (T_O - T_f) - m C_p' (T_i - T_f)$$

$$(T_i - T_f)$$

 $\Delta$ H sample i. e. the enthalpy decrease from the initial temperature to the final equilibrium temperature is obtained from a knowledge of the initial temperature of the sample and a graphical record of the temperature distribution measurement of the receiver. From this record of the temperature history the drift rate before and after the drop is obtained. Extrapolation of the drift rate back to a time where 60% of the receiver temperature rise has occurred yield the receiver temperature rise which would result if there had been no heat loss from the receiver system. The graphical method has been discussed extensively elsewhere (6,7,8) and a practice has formed to yield very reliable results.

The drop wire was of nickel chromium alloy 0.2 mm diameter of known specific heat and chosen so that the heat capacity would be as low as possible and a correction made for its effect.

The evaluation of the enthalpy change for any one drop can therefore be evaluated. Several drops have to be made on each sample to ensure uniformity of techniques and reproducibility of results to the order of  $\pm$  3% or better. Since the fina quilibrium temperature is never exactly the same for all cases the measured enthalpy for all the samples and all cases should be corrected to some chosen final temperature.

Since the specific heat of a material is the slope of the enthalpy temperature curve, a curve is made of the corrected enthalpy changes versus original temperature of the sample. The slope of the curve is evaluated graphically to yield the specific heat



at particular temperatures. Obviously the larger the number of data points (drops) obtained at different temperatures the higher the confidence in the final results due to the more accurate definition of the enthalopy temperature curve.

For the present materials the measurements to book by the adiabatic method had indicated that the different batches were very similar in property. It was decided therefore that instead of dropping all the samples from any one of the particular prechosen temperatures a more accurate record of the enthalpy temperature curve could be obtained by doubling the number of starting temperatures and halving the number of samples dropped at each temperature. This was the proceedure adopted and six or seven samples were dropped successively at least three times at any of the regular number of temperatures chosen within the approximate temperature ranges 500 to 1100K.

In all cases a very slow stream of dry pre-heated argon was introduced into the tube after it had been flushed several times with argon in order that the sample remained in a protected atmosphere prior to the drop. Each sample was weighed accurately prior to and after a drop as was the section of drop wire which was in the heated zone prior to the drop.

The measures taken to protect the sample worked satisfactorily until temperatures of the order of 1100K were obtained. At these temperatures the samples were found to degrade rapidly and attempts to attain temperatures of 1200K had to be abandoned because of this degredation.



#### Section 4

### RESULTS AND DISCUSSION

### 4.1 Thermal Conductivity and Electrical Resitivity

The experimental values of thermal conductivity and electrical resistivity are given in table IV and V respectively. These results are also illustrated collectively in Figures 2 and 3 respectively, where the numbered points refer to the respective sample number. The values shown have not been corrected for any changes in dimension due to expansion of the sample.

From the previous analysis of the instrument design  $^{(1)}$  and of the respective order of correction which had to be applied to particular data points the thermal conductivity values are believed to be accurate to better than  $\pm$  3% up to the order of 800K and  $\pm$  5% to 1200K. The electrical resistivity values are similarly believed to be accurate to better than  $\pm$  1% over the temperature range studied. Smooth curves can be drawn through each individual set of result for each sample to yield value of each property which are within within accuracy quoted. Values of thermal conductivity obtained from these different curves are given in Table VI.

There are some small differences in the overall values of thermal conductivity and electrical resistivity of the different batches of beryllium measured. There differences tend to diminish as the temperature increases. The experimental points do indicate some slight differences in the shape of the thermal conductivity versus temperature relationship. These differences, however, are within the experimental accuracy. The electrical resistivity extreme values as indicated by samples 2 and 9 respectively which differ by the order of 10% at room temperature become much closer together as the temperature increases.

The results obtained are in very good agreement with those obtained previously for some hot pressed beryllium material  $^{(1,2)}$  which were measured in a similar apparatus but only to the order of 900K. The present results have now extended the curve a further 400K increment in temperature and indicate that the general shape and form of the curve is regular and that the absolute values are higher than those obtained by a completely different method on two flat plate samples of different purities.  $^{(9)}$ 



The shape of the electrical resistivity versus temperature curve indicates that there is a definite change in slope above 800K. This is similar to the behavior found by other workers for resistivity. while a small inflection has been found in the specific heat curve at similar temperatures. The slope of the resistivity curve is changing more rapidly as the temperature exceed 1200K. The present values and this latter behaviour are also in general agreement with other measurements (10) but in the latter case the studies were made in different environments, namely a vacuum or a helium atmospheres. The author suggeste that the protective environment was to be preferred. On heating to 1300K or higher the present sample all degraded or contaminated to such an extent that no reliable repeat values could be obtained on cooling to room temperature. From the agreement obtained between the sample and the repeat measurement made after heating to the order of 1200K is felt that the values shown are real, certainly to the order of 1200K. However those above this temperature may possibly be affected by the onset of contamination.

Table VII contains values of thermal conductivity and electrical resistivity obtained from smooth curves drawn through all of the experimental points together with the derived Lorenz Function and lattice component of thermal conductivity. All thermal conductivity valeus are within  $\pm$  9% of the smooth curve and for the electrical resistivity they are within  $\pm$  5%. These values, while not accurate enough for the original purposes for which the measurments were made, are given to provide a basis upon which to calculate the thermal conductivity of other samples of hot pressed beryllium of similar purities and known electrical resistivity.

From Table VI it can be seen that the Lorenz Function decreases by less than 10% over the complete temperature range and the absolute value indicating that even at 1300K there is an approximate 20% lattice component of thermal conductivity. Figure 4 shows the approximate linear variation of lattice component with the reciprocal of the absolute temperature similar to that found by Powell (12) for some other type of beryllium material. The behavior of the present hot pressed material particularly of the  $\lambda$  g is somewhat different to that of Powell's materials.



### 4.2 Specific Heat

Values of specific heat obtained from smooth curves drawn through the record obtained for each sample measured in the adiabatic calorimeter are given in Table VIII. Table IX contains mean values of the measured enthalpy corrected to a standard final temperature of 24.5C together with the desired specific heat obtained graphically from an analysis of the enthalpy  $\underline{v}$  temperature relationship.

The specific heat results obtained by the adiabatic method are believed to be accurate to  $\pm 2\%$  which the enthalpy values obtained by the drop calorimeter are believed to be accurate to  $\pm 4\%$ .

The values obtained by the adiabatic method are seen to be in excellent agreement, the overall values being within  $\pm 1.5\%$  of the average value. Similarly the enthalpy results obtained were in excellent agreement as were the derived values of specific heat for temperatures of 300 and 350K where the property could be evaluated from the results of both methods would indicate that the results are within the accuracy quoted.

The mean values of specific heat obtained from all samples by both methods are shown in Figure 5. For comparison purposes the mean values of specific heat obtained at the National Bureau of Standards<sup>(11)</sup> for two pure samples of beryllium are also shown. The agreement is extremely good, the present values being only a maximum of 2% high over parts of the temperature ranges. Some small differences are likely to be present due to the differences in purity between the two samples. Above 1100K the results appear to fall to below the values obtained at NBS and it is believed that this is due to the fact that the material was becoming contaminated. No results were obtained at temperatures much in excess of 1100K due to subsequent degredation of the samples in the atmosphere provided.



### Section 5

### **SUMMARY**

The thermal conductivity of hot pressed beryllium has been measured on twelve samples of slightly different purities over the range 300 to 1200K. Small differences in this property and in electrical resistivity were found for individual samples but for samples of the same batches the values were in good agreement.

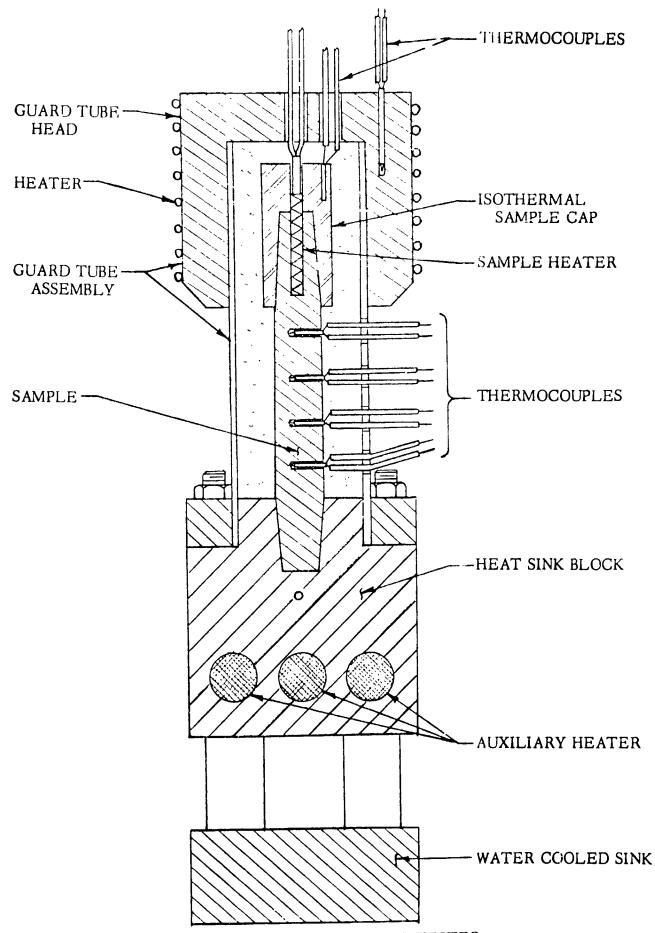
Specific heat measurement were carried out to the order of 1100K by a comination of adiabatic and drop calorimetery in thirteen samples. No really significant differences were found for the different batches.

The author wishes to thank Mr. J. Brazel of General Electric Company for his cooperation on their work and his colleague Mr. R.W. Hoyden, who carried out much of the experimental work described.



### REFERENCES

- 1. Dynatech Report No. 689, GEM 21, submitted to General Electric Company 028E-40732, 1 Jan. 1967.
- 2. Tye, R. P and Quinn, J. (1968), <u>Proceedings of the Fourth Symposiums on Thermal</u>
  Physical Properties. J. R. Moszynski, (Ed.) ASME, New York.
- 3. Hoagland, L.C., de Winter, F. and Reti, A.R. (1967), Trans. ISA 6 (2) 111-121.
- 4. Ginnings, D.C. and Corruccini, R.J., (1947) J. Res. NBS 38 53 and 38 593.
- 5. Conway, J. B., Hein, R. A., (1965), Advances in Thermophysical Properties at Extreme Temperatures and Pressures, ASME, New York
- 6. Coops, J., Jessup, R.S. and van Nes, K., (1956) in Rossini, F.D., Experimental Thermochemistry, Interscience, New York.
- 7. Sturtevant, J. M., (1959) in Weissberger, A., Physical Methods in Organic Chemistry Part I, 3rd Ed. Intersceience, New York.
- 8. Wadsö, I., (1966) Science Tools 13, 33 39.
- 9. Fieldhouse, I. B., Hedge, J. C., et al, (1958) WADC Tech Rept. No. 57-487, ADISO 954.
- 10. Lampson, F. K., USAEC Publication NEPA 1860 9 pp.
- 11. Ginnings, D.C., Douglas, T.B. and Ball, Anne F., (1951) J. Am. Chem. Soc., 73, 1236 40.
- 12. Powell, R.W., (1953) Phil. Mag. 44, 645-663.



DYNATECH ABSOLUTE AXIAL ROD TESTER THERMAL CONDUCTIVITY APPARATUS

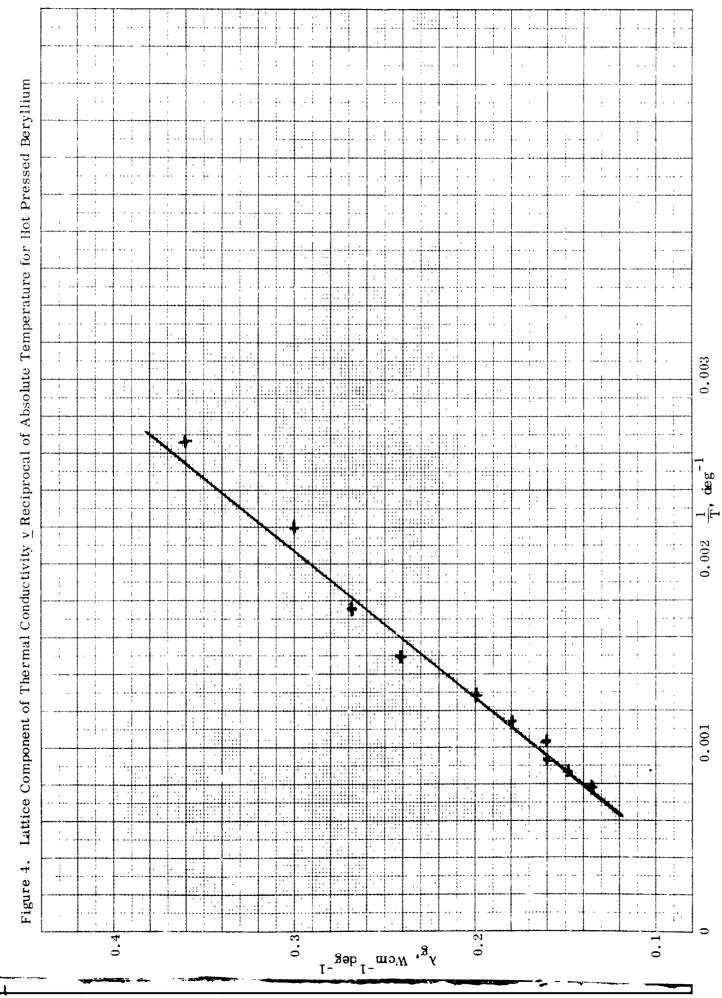
Figure 1

MAS 1913 FO THE CENTIMETER 48 1913

1250 $1\dot{0}00$ Temperature, K Electrical Resistivity of Hot Pressed Beryllium 750Figure 3. 45 20 15 35

KE 10 Y TO THE CENTIMETER 46 1513

KELFFELS ESSIRCY



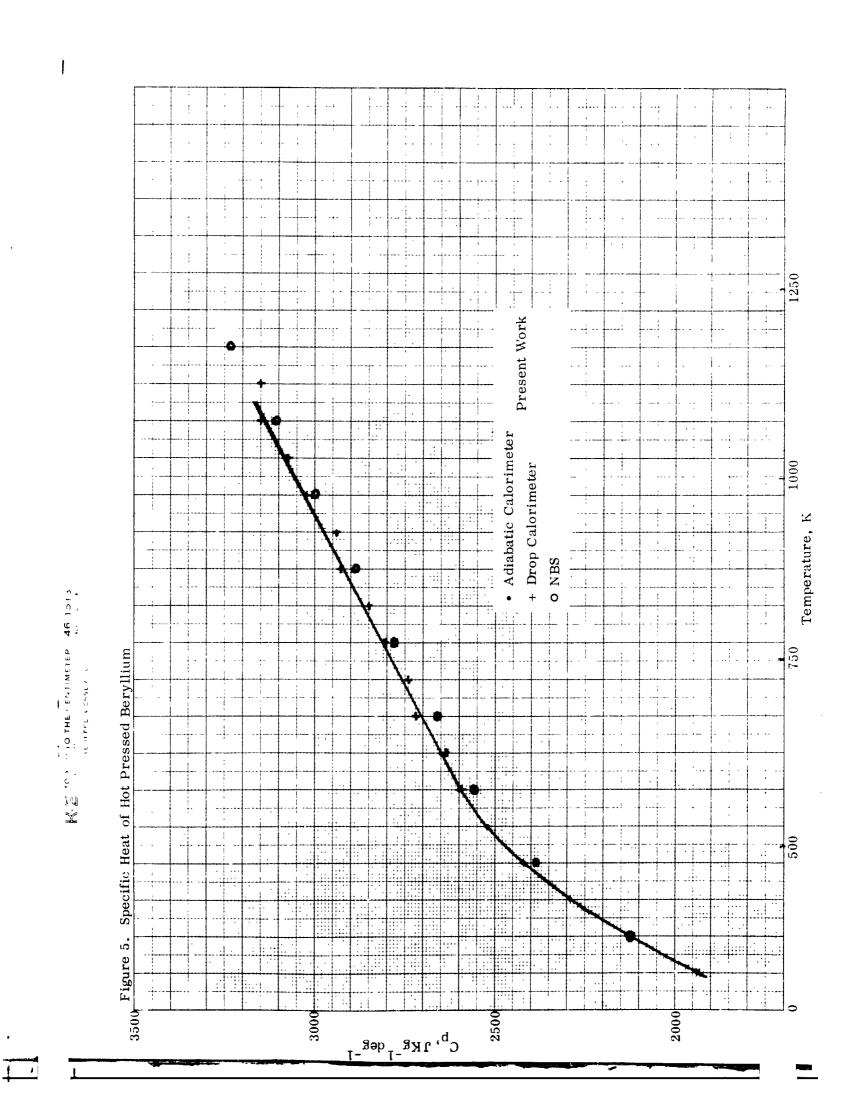


Table IV

Measured Values of Thermal Conductivity,  $\lambda$ , W cm<sup>-1</sup> deg<sup>-1</sup>, at Various Mean Temperatures. T, °C, For Twelve Samples of Hot Pressed Beryllium

T #1	<	T #2	~	£# T	~	T #4	~	T #5		9# L	\ \ \ \
68	1.83	70.5	1.89	77.5	1.79	11.7	1.69	59.5	1.84	88	1.71
272	1.40	509	1.53	275	1.42	320.5	1.38	299	1.36	312.5	1,33
476.5	1.17	402	1, 22	069	1.01	504	1.11	483.5	1.18	525	1.16
702	1.00	646.5	1.03	882.5	0.88	789	0.91	725	0.96	692	0.98
892.5	0,83	910	0.89	454	1.13	920	0.83	893	0.8	913. 7	0.92
432.5	1.18	388	1.34	1022	0.79	1007	0.84	511	1.08	202.5	1.51
896	0.86	960.5	0.85				<del></del>			1028	0.81
L# T	ų	L #8	~	6# L	~	T #10	~	T #11	~	T #12	سہ
252	1.42	104	1,69	77.5	1.76	125	1.68	82	1.72	275	1.39
450.5	1, 11	289	1.37	253	1.46	342	1.29	343	1.27	122	1.66
74	1.73	431.5	1.22	512.5	1.05	465.5	1.21	541.5	1.16	455.5	1.20
398.5	1.26	618	1.05	687	0.96	735	1.02	762.5	0.91	651	1.01
685	1.00	829	0.98	911	0.92	979	0.88	883	0.94	942	0.81
872	0.84	977	0.85	422.5	1.20	2805	1.40	973.5	0.82	830.5	0.97
1004	0.86			1011	0.83	606	0.85			926	0.88
											-

Table V Measured Values of Electrical Resistivity,  $10^6 \rho$ . ohm cm, at Various Mean Temperatures T, °C For Six Samples of Hot Pressed Beryllium

$10^6 \rho$ T $10^6 \rho$ T $10^0 \rho$ T $24.3$ $4.72$ $24.3$ $4.72$ $24.3$ $4.72$ $24.3$ $4.72$ $24.3$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$ $4.72$	#5	,	#		9#	¢	8#	c	6#	·	#11	,
1       4.05       22.2       4.50       25.6       4.61       22       4.25       24.3       4.72         5       6.99       98.3       6.51       101.3       6.88       123       7.1       120       7.4         3       11.22       276       12.32       357       15.15       233.3       10.74       227.5       10.95         15.53       368.5       15.20       496       20.42       341.2       14.42       392.5       10.95         21.3       591.3       24.09       635       25.72       473.3       18.93       603.3       25.15         24.83       737.1       29.92       726       30.12       612.2       24.52       686.3       25.15         36.2       885       39.1       848       37.1       699.9       28.12       772.5       32.4         35.6       976       44.1       985       44.9       840.5       35.5       860       38.3         36.14.88       37.1       699.9       25.1       46.3       935       45.6         44.2       36.4       36.1       36.2       45.6       46.9       46.9       46.9       46.9       46.9	T	$10^6   ho$		$10^{6}\rho$		$10^{6}\rho$		$10^{0}\rho$	Τ	$10^{6}\rho$	T	$10^{0} \rho$
5         6.99         98.3         6.51         101.3         6.88         123         7.1         120         7.4           3         11,22         276         12.32         357         15.15         233.3         10.74         227.5         10.95           15.53         368.5         15.20         496         20.42         341.2         14.42         392.5         16.95           21.3         591.3         24.09         635         25.72         473.3         18.93         603.3         25.15           24.83         737.1         29.92         726         30.12         612.2         24.52         686.3         28.81           30.22         885         39.1         848         37.1         699.9         28.12         772.5         32.4           35.6         976         44.1         985         44.9         840.5         35.5         860         38.3           5         14.88         36.6         45.3         935         42.2           5         14.88         36.6         45.3         935         45.6           44.2         44.2         45.3         995         45.6           46.9 <t< th=""><td>22.1</td><td>4.05</td><td>22.2</td><td>4.50</td><td>25.6</td><td>4.61</td><td>22</td><td>4, 25</td><td>24.3</td><td>4.72</td><td>24.5</td><td>4.70</td></t<>	22.1	4.05	22.2	4.50	25.6	4.61	22	4, 25	24.3	4.72	24.5	4.70
3         11,22         276         12.32         357         15.15         233.3         10,74         227.5         10.95           15,53         368.5         15.20         496         20,42         341.2         14.42         392.5         16.95           21.3         591.3         24.09         635         25.72         473.3         18.93         603.3         25.15           24.83         737.1         29.92         726         30.12         612.2         24.52         686.3         25.15           5         30.22         885         39.1         848         37.1         699.9         28.12         772.5         32.4           5         40.66         44.1         985         44.9         840.5         35.5         860         38.3           5         14.88         37.1         10.18         1003         45.3         935         42.2           44.2         44.2         45.9         840.5         35.5         995         45.6           46.9         46.9         45.3         995         45.6         45.6	122.5	6. 99	98.3	6.51	101.3	6.88	123	7.1	120	7.4	123	7.43
15.53       368.5       15.20       496       20.42       341.2       14.42       392.5       16.95         21.3       591.3       24.09       635       25.72       473.3       18.93       603.3       25.15         24.83       737.1       29.92       726       30.12       612.2       24.52       686.3       28.81         5       30.22       885       39.1       848       37.1       699.9       28.12       772.5       32.4         5       40.66       44.1       985       44.9       840.5       35.5       860       38.3         5       14.88       10.64       10.18       1003       45.3       935       45.6         44.2       44.2       44.2       10.18       1003       45.3       995       45.6	253.3	11.22	276	12.32	357	15.15	233.3	10.74	227.5	10.95	222	10.82
21.3       591.3       24.09       635       25.72       473.3       18.93       603.3       25.15         24.83       737.1       29.92       726       30.12       612.2       24.52       686.3       28.81         5       30.22       885       39.1       848       37.1       699.9       28.12       772.5       32.4         5       40.66       44.1       985       44.9       840.5       35.5       860       38.3         5       40.66       7       10.18       1003       45.3       935       42.2         44.2       44.2       44.2       45.3       995       45.6         46.9       46.9       45.6       45.6       45.6	404	15, 53	368.5	15.20	496	20.42	341.2	14.42	392.5	16.95	353.5	15.03
5         30.22         885         39.1         848         37.1         699.9         28.12         772.5         32.4           35.6         976         44.1         985         44.9         840.5         35.5         860         38.3           5         40.66         36.0         44.1         10.18         10.03         45.3         935         42.2           5         14.88         44.2         36.6         46.9         46.9         46.9         45.3         995         45.6	539	21.3	591.3	24.09	635	25.72	473.3	18.93	603.3	25.15	505.2	20.41
5       30.22       885       39.1       848       37.1       699.9       28.12       772.5       32.4         35.6       976       44.1       985       44.9       840.5       35.5       860       38.3         5       40.66       210.4       10.18       1003       45.3       935       42.2         5       14.88       44.2       46.9       10.18       10.18       10.18       10.6       46.3       19.6         46.9       46.9       46.9       45.6       45.6       45.6       45.6       46.9	627	24.83	737.1	29.92	726	30.12	612.2	24.52	686.3	28.81	616	24.83
35.6       976       44.1       985       44.9       840.5       35.5       860       38.3         .5       40.66       210.4       10.18       1003       45.3       935       42.2         .5       14.2       44.2       995       45.6       995       45.6	739.5	30.22	885	39.1	848	37.1	6.669	28.12	772.5	32.4	834.5	36.2
.5       40.66       210.4       10.18       1003       45.3       935       42.2         .5       14.88       46.3       19.6         44.2       995       45.6	826	35.6	926	44.1	985	44.9	840.5	35.5	860	38.3	899	40.9
.5       14.88       46.3         44.2       995         46.9       995	900.5	40.66			210.4	10.18	1003	45.3	935	42.2	993	44.8
44.2 995	362. 5	14.88				-			466.3	19.6		
	973	44.2							995	45.6		
	1010	46.9										

 $\label{eq:table VI} Thermal\ conductivity\ \lambda,\ W\ cm^{-1}\ deg^{-1}\ for\ twelve\ samples\ of\ hot\ pressed\ beryllium\ .$   $\ Values\ obtained\ from\ smooth\ curves\ drawn\ through\ experimental\ points$ 

Sample #		λ	W cm <sup>-1</sup> d	eg - 1		
Temperatur'.	373K	573K	773K	973K	1173K	1273K
1	1.76	1.38	1. 15	0.97	0.88	[0,83]
2	1.77	1.38	1.15	0.98	0.89	[0.85]
3	1.75	1.36	1.12	0.96	0.86	0.82
4	1.75	1.37	1.13	0.96	0.85	0.84
5	1.76	1.36	1.12	0.95	0.83	
6	1.69	1.37	1.16	1.0	0.90	0.86
7	1.68	1.35	1, 13	0.99	0.86	[0.82]
8	1.69	1.37	1.15	1.0	0.89	[0.85]
9	1.71	1.38	1.16	1.0	0.90	[0.87]
10	1.70	1.38	1.16	1.0	0.90	[0.85]
11	1.67	1.36	1. 14	0.99	0.88	0.84
12	1.69	1.37	1, 16	0.99	0.89	[0.86]

Values [ ] are extrapolated.

Table VII

Values of  $\lambda$  and  $\lambda_g$  ,W cm $^{-1}$  deg $^{-1}$ ,  $10^6~\rho$ , ohm cm, together with derived  $10^8$  Lorenz function,  $v^2$  deg $^{-2}$  at regular intervals of absolute temperature for hot pressed beryllium

Temperature, K	$10^6\rho$	λ	$^{\lambda}\mathrm{g}$	10 <sup>8</sup> L
373	6.7	1.72	0.355	3.09
473	9.6	1.51	0.30	3.07
573	12.8	1.37	0.275	3.06
673	16.4	1. 25	0.245	3.05
773	20.0	1. 15	0.20	2.98
873	24.4	1.06	0.18	2.96
973	28.7	0.99	0.16	2.93
1073	34.2	0.93	0.16	2.96
1173	39.9	0.87	0.15	2.95
1273	45.5	0.82	0.135	2.93

Table VIII

Values of Specinic heat,  $C_p$ ,  $J K_g^{-1} deg^{-1}$ , at regular intervals of temperature T. °C. obtained from smooth curves drawn through the experimental records.

Ç					ζ	1 - 1	1-27						
Sample					, p	b, J n deg	1 840						
**	1	C1	က	4	5	9	7	œ	6	10	111	12	13
20	1920	1905	1935	1940	1950	1945	1970	1950	1970	1970	1915	1920	1910
100	2120	2105	2120	2130	2140	2140	2145	2145	2140	2135	2100	2110	2100
150	2265		2285	2310	2305	2320	2325	2320	2310	2310	2255	2260	2255
200	2410	2405	2420	2430	2430	2435	2450	2460	2445	2455	2385	2395	2375
250	2510	2510	2510	2515	2535	2535	2550	2570	2540	2550	2490	2500	2480
300	2580	2595	2595	2605	2615	2615	2620	2630	2595	2610	2588	2595	2570
350	2640	2630	2630	2640	2665	2660	2660	2675	2660	2635	2675	2570	26: 5

TableIX

Experimental values of measured enthalpy ( $H_t - H_t$ ), J  $K_g^{-1}$  and derived specific heat,  $C_p$ , J  $K_g^{-1}$  deg<sup>-1</sup> temperatures T. °C for thirteen hot pressed beryllium samples.

(All values corrected to a final temperature  $T_f = 24.5^{\circ}C$ )

T	No. of different samples used	H <sub>t</sub> - H <sub>24.5</sub>	Spread of Data	Т	Ср
259.3	7	513,000	± 3.7	300	2595
316.1	7	667,000	± 2.2	350	2635
377.4	6	822,000	± 1.9	400	2720
423.6	7	951,000	± 1.1	450	2740
479.9	6	1,100,000	+ 1.3	500	2805
<b>53</b> 7. 2	7	1,265,000	+ 1.6	550	2850
597.0	6	1,451,000	÷ 1.2	600	2930
640.1	5	1,576,000	± 1.6	650	2940
698.2	6	1,751,000	± 2.0	700	3025
752.7	6	1,372,000	± 1.7	750	3075
804.0	5	2,078,000	: 2.7	800	3150
837. 5	5	2,170,000	± 4.3	850	3150